

Chemical constituents of *Centaurea omphalotricha* Coss. & Durieu ex Batt. & Trab.

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Abstract: The investigation of the aerial parts of *Centaurea omphalotricha* Coss. & Durieu ex Batt. & Trab. allowed the isolation of nine secondary metabolites corresponding to five flavonoids: oroxylin A (**1**), chrysin (**2**), tenaxin II (**3**), 5,7,2'-trihydroxyflavone (**4**) and quercetin (**5**), and four triterpenoids: lupeol (**6**), taraxasterol (**7**), daucosterol (**8**) and β -sitosterol (**9**). Their structures were established by spectroscopic methods such as ¹H and ¹³C NMR, COSY, HSQC, and HMBC experiments, and ESI-MS, and comparison with literature data. The flavonoids tenaxin II (**3**) and 5,7,2'-trihydroxyflavone (**4**) are new for the genus *Centaurea* L.

Keywords: *Centaurea omphalotricha*; Asteraceae; Flavonoids; Triterpenoids.

1. Plant Source

Centaurea omphalotricha Coss. & Durieu ex Batt. & Trab. belongs to the genus *Centaurea* L. of the family Asteraceae [1,2]. This genus comprises about 500 species from which 50 are growing spontaneously in Algeria [3]. *C. omphalotricha* Coss. & Durieu ex Batt. & Trab. an endemic species for Algeria and Tunisia, is a perennial plant, 40-50 cm long, with yellow colored flowers, localized especially in the desert regions [3].

The plant material was collected in May 2009 in the vicinity of Biskra (Oued Biskra, Algeria) and was identified by Pr. Bachir Oudjehih, Agronomic Institute of the University of Batna. A voucher specimen is kept under the number 590/LCCE.

2. Previous Studies

To the best of our knowledge, no phytochemical and biological works have been reported on *C. omphalotricha* Coss. & Durieu ex Batt. & Trab.

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3. Present Study

Isolated compounds were characterized by UV (Beckman DU-600), IR (KBr, Shimadzu IR-470 and Jasco FT/IR-4100), positive and negative ESI-MS (ion trap Bruker Esquire) and extensive 1D and 2D NMR analysis (COSY, HSQC, HMBC, Bruker Avance Spectrometer, ^1H 500 MHz, ^{13}C 125 MHz). Optical rotations were measured on a Perkin-Elmer 241 polarimeter. CC was carried out on Kieselgel 60 (320-400 mesh) and Sephadex LH-20. Analytical and preparative (1 mm thickness) TLCs were carried on silica gel (Kieselgel 60 F₂₅₄, Merck) and RP-18 (Kieselgel 60 F_{254S}) plates.

In this study, we have isolated nine known compounds including five flavonoids and four triterpenoids (Figure 1) from the CH_2Cl_2 extract of the aerial parts of *C. omphalotricha*. They were identified unambiguously by comparison of their NMR and mass spectra, and values of optical rotations with published data as oroxylin A (**1**) [4], chrysin (**2**) [5], tenaxin II (**3**) [6], 5,7,2'-trihydroxyflavone (**4**) [7], quercetin (**5**) [8], lupeol (**6**) and taraxasterol (**7**) [9], daucosterol (**8**) [10] and β -sitosterol (**9**) [11]. All of the isolated compounds were characterized by using spectroscopic methods especially 1D and 2D NMR and mass spectrometry ESI.

Powdered dried of aerial parts (800 g) from *C. omphalotricha* were extracted with 70% ethanol during 3 days at room temperature. The ethanol extract was concentrated. The concentrate was taken up with different solvents with increasing polarity: petroleum ether, dichloromethane, ethyl acetate and *n*-butanol, successively. Filtration and evaporation to dryness gave 3.1 g of petroleum ether, 5.8 g of dichloromethane, 4.7 g of ethyl acetate and 27.64 g of *n*-butanol extracts. The CH_2Cl_2 extract (5 g) was subjected to vacuum liquid chromatography (VLC) performed over silica gel (50 \times 50 mm; fractions of 100 mL) using a gradient of petroleum ether/EtOAc (100:0 to 0:100) and EtOAc/MeOH (100:0 to 60:40). Fractions having similar TLC profiles were pooled to afford 11 fractions. Fractions F-3 and F-4 were grouped and submitted to silica gel column chromatography eluting with CHCl_3 to provide two fractions. The first fraction was purified using Sephadex LH-20 CC and elution with CHCl_3 to lead 12 mg of lupeol (**6**), while the second one was precipitated in EtOH to afford 4.2 mg of taraxasterol (**7**). Fractions F-5 and F-6 mixed were applied to silica gel CC eluting with $\text{CHCl}_3/\text{MeOH}$ (100:0 to 80:20) to obtain 10 fractions. Fractions eluted with $\text{CHCl}_3/\text{MeOH}$ (99:1) were purified by TLC RP-18 to give 8 mg of β -sitosterol (**9**). Fractions F-7 and F-8 were combined and precipitated in petroleum ether to furnish a yellow powder which was submitted to silica gel CC eluting with $\text{CH}_2\text{Cl}_2/\text{acetone}$ (100:0 to 90:10) to afford 11 fractions. Fractions eluted with CH_2Cl_2 contained 25 mg of oroxylin A (**1**). Preparative TLC of fractions eluted with $\text{CH}_2\text{Cl}_2/\text{acetone}$ (99.5:0.5), developed with a mixture of $\text{CHCl}_3/\text{MeOH}$ (95:5), allowed isolation of chrysin (**2**) (17 mg). Precipitation of fractions eluted with $\text{CH}_2\text{Cl}_2/\text{acetone}$ (99:1) in a mixture of CH_2Cl_2 with small amount of acetone yielded tenaxin II (**3**) (5 mg). Purification of fractions eluted with $\text{CH}_2\text{Cl}_2/\text{acetone}$ (95:5) by precipitation in acetone gave 6.5 mg of 5,7,2'-trihydroxyflavone (**4**). The filtrate of fractions F-7 and F-8 containing a major product was precipitated in a mixture of $\text{CH}_2\text{Cl}_2/\text{acetone}$ to provide 10 mg of quercetin (**5**). Fraction F-10 was subjected to Sephadex LH-20 CC eluting with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (100:0, 99:1, 93:3, 95:5) to afford 6 fractions. Fractions eluted with $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (99:1 and 93:3) were mixed and precipitated in acetone to obtain 18.5 mg of daucosterol (**8**).

Centaurea omphalotricha Coss. & Durieu ex Batt. & Trab. belongs to the family Asteraceae which contains divers secondary metabolites like flavonoids [12], triterpenoids [13] and sesquiterpene lactones [14]. This investigation allowed isolation of flavonoid aglycones such as oroxylin A (**1**), chrysin (**2**) and quercetin (**5**) which occur in several other *Centaurea* species like *C. pseudoscapiosa* [15], *C. scabiosa* [16], *C. malacitana* [17] and *C. napifolia* [18] as well as commun triterpenoids like lupeol (**6**), taraxasterol (**7**), daucosterol (**8**) and β -sitosterol (**9**). It is important to indicate that flavonoids tenaxin II (**3**) and 5,7,2'-trihydroxyflavone (**4**) isolated previously from *Scutellaria* species [6,7] were found for the first time in *Centaurea* genus.

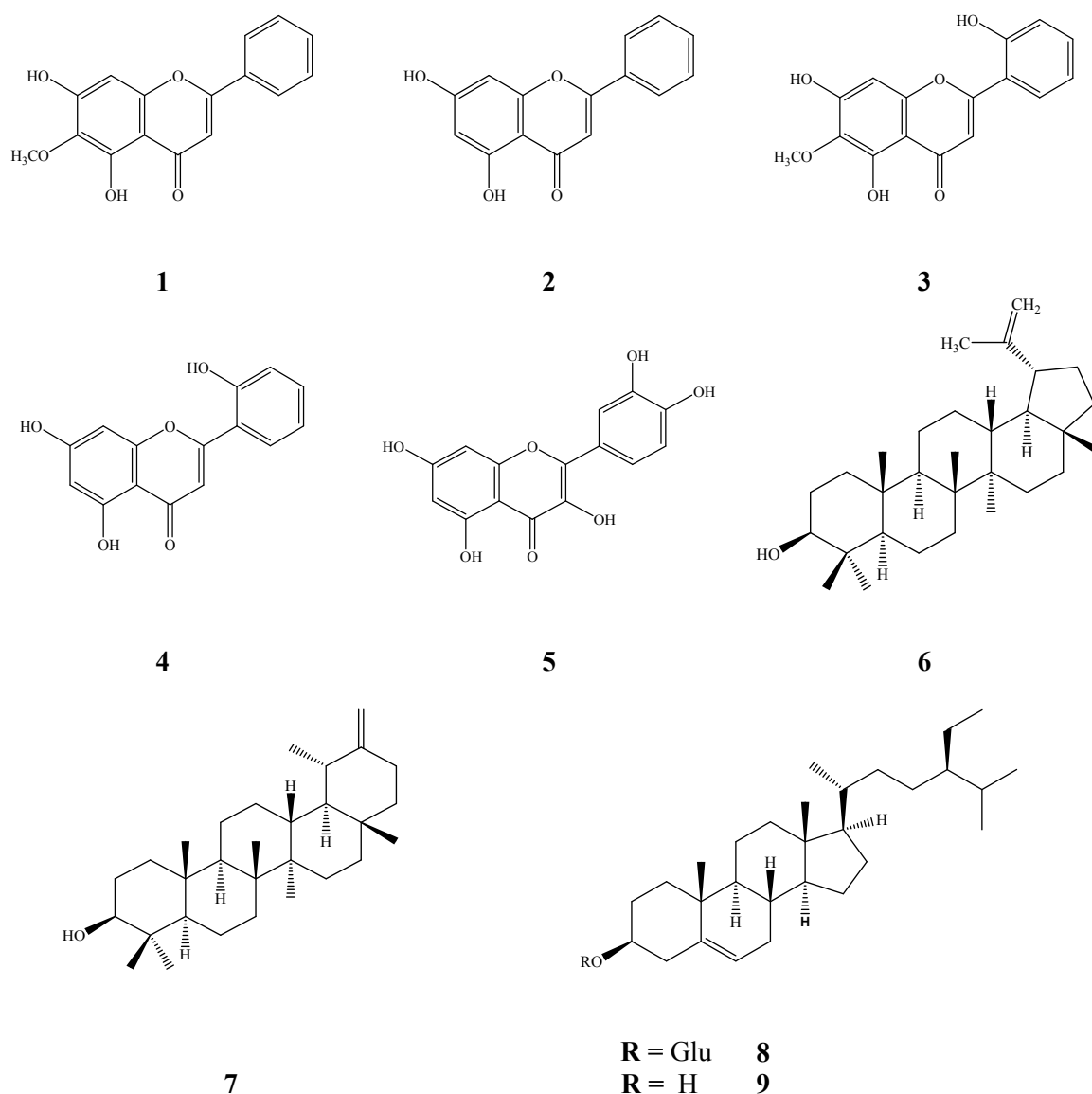


Figure 1. Structures of the isolated compounds 1-9

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Supporting Information

Supporting information accompanies this paper on <http://www.acgpubs.org/RNP>

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